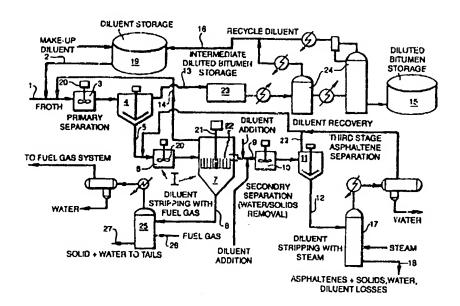
OPIC OPIC CIPO CANADIAN INTELLECTUAL PROPERTY OFFICE

(12) (19) (CA) Demande-Application

(21)(A1) 2,232,929

(22) 1998/03/24 (43) 1998/09/25

- (72) BIRKHOLZ, Robert Karl Otto, CA
- (72) POWER, William James, CA
- (72) TIPMAN, Norman Robert, CA
- (71) Shell Canada Limited, CA
- (51) Int.Cl.6 B03B 9/02
- (30) 1997/03/25 (2200899) CA
- (54) METHODE DE TRAITEMENT D'UNE MOUSSE DE SABLE BITUMINEUX DILUEE
- (54) METHOD FOR PROCESSING A DILUTED OIL SAND FROTH



(57) Méthode de traitement d'une mousse de sable bitumineux extraite sous forme de flux de sousverse (5) à partir d'une cuve primaire de séparation de mousse de bitume (4), comprenant l'agitation vigoureuse de la sousverse dans un réservoir d'agitation (6), à un niveau de cisaillement tel que sont formés des agglomérés d'asphaltène et d'argile alors que la redispersion des agglomérés ainsi formés demeure limitée, et la séparation desdits agglomérés sous forme de flux de mixtes (9), des autres composants, par exemple dans une cuve de séparation secondaire (7), dans laquelle la mousse est lentement agitée.

(57) A method for processing an oil sand froth that is withdrawn as an underflow stream (5) from a primary bitumen froth separation vessel (4) comprises the steps of vigoreusly agitating the underflow in an agitating tank (6) at such a shear level that agglomerates of asphaltenes and clays are formed whilst redispersion of the thus formed agglomerates remains limited and separating said agglomerates as a middlings stream (9) from the other components e.g. in a secondary separation vessel (7) in which the froth is gently agitated.

TS 9133

ABSTRACT

METHOD FOR PROCESSING A DILUTED OIL SAND FROTH

A method for processing an oil sand froth that is withdrawn as an underflow stream (5) from a primary bitumen froth separation vessel (4) comprises the steps of vigorously agitating the underflow in an agitating tank (6) at such a shear level that agglomerates of asphaltenes and clays are formed whilst redispersion of the thus formed agglomerates remains limited and separating said agglomerates as a middlings stream (9) from the other components e.g. in a secondary separation vessel (7) in which the froth is gently agitated.

(FIG. 1)

C36/TS9133FF

METHOD FOR PROCESSING A DILUTED OIL SAND FROTH

The invention relates to a method for processing a diluted oil sand froth.

More particularly, the invention relates to a method for processing a diluted oil sand froth that contains hydrocarbons, water, clays and coarse solids and that is withdrawn from a lower part of a primary fluid separation vessel of a paraffinic solvent froth treatment unit in an oil sand processing plant.

5

10

15

20

25

In such a plant mined oil sand is mixed with water to produce a sturry whereupon the slurry is screened to remove oversize rocks and oil sand lumps. The thus generated pumpable slurry is subsequently conditioned by the combined action of selectively wetting the sand, softening of the hydrocarbons and mechanical agitation and then fed to a bitumen extraction process.

The bitumen froth produced from the bitumen extraction process is mixed with a paraffinic diluent or solvent and fed to a primary separation vessel to initiate inversion of the emulsion. This will cause dilute bitumen globules to rise and form a dilute bitumen upper layer. This dilute bitumen is subsequently withdrawn from the overflow of the vessel and further treated.

The other components of the inverted emulsion which separates into a dense fluid, containing water, solids, clays and hydrocarbon emulsion enriched in asphaltenes are withdrawn as an underflow from the lower part, usually the bottom, of the primary separation vessel. These components are then usually further treated in a

secondary separation vessel and/or a cyclone or a decanter centrifuge to separate the water/solids phase from the asphaltenes, bitumen and diluent.

5

10

15

20

25

30

Canadian patent specification No. 940,853 discloses a tar sand processing method in which a propane-butane solvent is mixed with tar-oil froths to make an emulsion that can be broken and separated in a controlled fashion.

The specification describes a rag layer with agglomerated asphaltics formed between the lower layer of water and solids and the upper layer of hydrocarbon and solvent when the ratio of solvent to oil is sufficiently high (page 3, lines 9 to 31). The known solvent is restricted to propane-butane combinations. The teachings do not include any direction on stirring to promote agglomeration of asphaltenes and clays in the underflow.

Further, an article entitled "Measurement of asphaltene flocculation in bitumen solutions" published in the Journal of Canadian Petroleum Technology, October 1986, Volume 25, No. 5, pages 33-37 describes a series of tests in which solvent addition to bitumen triggers asphaltene precipitation.

US patent specifications 4,765,885, 4,891,131 and 5,017,281 disclose the use of a separation tank in which ultrasonic sound waves are transmitted to separate an aqueous tar sand slurry into a liquid hydrocarbon oil fraction which rises to the top of the suspension and to cause asphaltenes and preasphaltenes to form charcoal-like agglomerates which settle to the bottom of the tank. The known tank is equipped with a mixing blade which is rotated during the separation process but which generates only a relatively low level of turbulence such that gravity separation still takes place.

US patent specification No. 4,906,355 discloses a tar sand extraction process in which a tar sand slurry is mixed with specific solvents having limited solubility for asphaltenes, notably C3-C7 alkanes and corresponding petroleum fractions, to form agglomerates of asphaltenes and mineral fines. A substantial portion of these agglomerates are subsequently gravitationally separated in a lamella separator to avoid substantial attrition of the agglomerates.

5

10

15

20

25

30

A problem encountered with the known separation systems is that asphaltenes and fine clays are difficult to remove from the slurry, so that in order to achieve an acceptable purity of any waste water and tailings, complex and expensive waste water and tailings cleaning facilities are required.

The present invention aims to provide a method for treating the underflow of a paraffinic solvent primary separation vessel such that any asphaltenes and clays can be removed in an efficient and effective manner.

The method according to the invention thereto comprises the steps of:

- agitating the underflow stream at such a shear level that agglomerates of asphaltenes and clays are formed whilst dispersion of the thus formed agglomerates remains limited;
- adding paraffinic solvent to remove entrained bitumen;
- separating at least a substantial portion of said agglomerates from the other components of the underflow scream; and
- withdrawing the thus separated agglomerates as a separate product stream.

5

10

15

20

25

30

Preferably the underflow stream is agitated by an impeller which is located in an agitating tank and which is driven at a power rate which is between 0.5 and 5 kW per $\rm m^3$ of the volume of the agitating tank.

It is also preferred that the agitated underflow stream flowing from the agitating tank has solvent added to it and is fed into a secondary separation vessel in which the fluid mixture is agitated at a lower shear rate than in the agitating tank, which lower shear rate is low enough to maintain the agglomerates of asphaltenes and clays within an intermediate fluid layer but high enough to release other hydrocarbons into an upper fluid layer and to release water and coarse solids into a lower fluid layer within said vessel and which vessel is equipped with a lower fluid outlet through which at least a substantial proportion of water and coarse solids are removed from the vessel, an intermediate fluid outlet through which at least a substantial proportion of said agglomerates of asphaltenes and clays are withdrawn from the vessel and an upper fluid outlet through which at least a substantial proportion of other hydrocarbons are removed from the vessel.

In that case it is preferred that the underflow stream is gently agitated in the secondary separation vessel by means of a stirring device which is slowly rotated at a power rate which is between 0.1 and 0.5 kW per m³ of volume of the secondary separation vessel.

These and other features, objects, aspects and advantages of the method according to the invention are disclosed in the accompanying claims, abstract, drawings and detailed description with reference to the drawings.

The invention will now be described in more detail with reference to the accompanying drawings, in which:

Fig. 1 depicts a schematic flow-scheme of a paraffinic solvent froth treatment plant in which arrows I point at an agitating tank and secondary separation vessel in which the method according to the invention is applied; and

5

10

15

20

25

30

Fig. 2 depicts a schematic flow-scheme of an alternative paraffinic solvent froth treatment plant in which the middlings and tailings from the secondary separation vessel are combined and mixed with fresh solvent. The resultant mixture is separated in a third stage separation vessel.

Referring now to Fig. 1, stream 1 represents a stream of bitumen froth comprising a mixture of bitumen, water and solids to which a stream 2 of paraffinic diluent and a recycle everflow stream 14 containing hydrocarbon and diluent from secondary separation vessel 7 are added to typically achieve a ratio of about two volumes of diluent per volume of bitumen in the slurry stream 1.

The diluent and slurry are subsequently mixed in a mixing tank 3 and fed to a primary separation vessel 4.

The bitumen froth stream 1 typically contains 25-30% by weight water, some of which is free water carried over to the froth during extraction, and approximately 5% of the water which is in the form of a micro-emulsion in the bitumen with droplet sizes of 2-10 microns. Mixing of this froth with the above-mentioned quantity of paraffinic diluent in the mixing tank 3 results in the destabilization of the emulsion and the subsequent formation of a rag layer at the hydrocarbon/water interface in the primary separation vessel 1. Furthermore, the diluent/froth ratio is above the inversion point of the emulsion which results in the precipitation of more asphaltenes, with the resulting

reduction of asphaltenes in the hydrocarbon product overflow stream 13 from the primary separation vessel.

5

10

15

20

25

30

}

The underflow 5, containing water, solids and hydrocarbon emulsion enriched in asphaltenes, is withdrawn from the conical bottom of the primary separation vessel 4 and fed into an agitating tank 6 in which an impeller 20 agitates the fluid mixture at such a shear level that agglomerates of asphaltenes are formed out that redispersion of the thus formed agglomerates remains limited. To achieve this relatively high shear level the impeller 20 is typically rotated at a power rate of between 0.5 and 5 kW per m³ of volume of the agitating tank 6. In the agitated underflow small asphaltene agglomerates are formed in which the very fine clays present in the emulsion are co-absorbed.

The agitated underflow stream is subsequently fed into the secondary separation vessel 7 which is designed to provide sufficient retention time to allow a hydrocarbon phase to be recovered from the top of the vessel 7 as an overflow stream 14 comprising mainly hydrocarbon and diluent and for a mainly asphaltene agglomerates containing middle layer to be formed and to be discharged as an asphaltene-rich middlings stream 9 from the middle of the vessel and an underflow 8 of water and solids which is relatively hydrocarbon-free to be discharged from the bottom of the vessel 7. Overflow stream 14 is combined with bitumen froth stream 1 and diluent stream 2 and is subsequently fed into mixing tank 3.

A stirring device 21, inside vessel 7, agitates the middle layer at such a shear level to encourage the asphaltene-rich middle layer to agglomerate and release water and solids into the lower fluid layer and

5

10

15

20

25

30

hydrocarbons and diluent into the upper fluid layer. To achieve this relatively low shear, the stirring device is rotated at a power rate between 0.1 and 0.5 kW per m^3 of volume of the secondary separation vessel.

The middlings stream 9 is combined with additional diluent and is subsequently fed to a third stage mixing tank 10 and asphaltene separator vessel 11 to disperse asphaltene agglomerates, recover entrained bitumen and to promote diluent recovery in a steam stripping unit 17 in which the asphaltene-rich underflow stream 12 from the third-stage separation vessel 11 is treated.

The overflow stream 22 of the third stage separation vessel 11 is combined with the underflow stream 5 of the primary separation vessel 4 and fed into agitating tank 6. The overflow stream 13 of the first separation vessel 4 is fed into an intermediate diluted bitumen storage tank 23. This overflow stream is then processed in a two-stage diluent recovery flash unit 24 in which diluent is flashed and thereby separated from the bitumen. Subsequently a diluted bitumen underflow stream is discharged into a diluted bitumen storage tank 15 and a recycled diluent overflow stream 16 is discharged into the diluent storage tank 19 which is the diluent supply source for the diluent stream 2 that is mixed with the bitumen froth stream 1.

The underflow stream 8 of the secondary separation vessel 7, which stream 8 mainly comprises coarse solids and water, with small quantities of hydrocarbon and asphaltenes is fed into a stripping unit 25 in which fuel gas is added to remove any small quantity of diluent from the solids and waste water stream 27.

The schematic flow scheme depicted in Fig. 2 is identical to that depicted in Fig. 1 except that the

underflow stream 8 from the secondary separation vessel 7 is combined with the asphaltenes rich middlings stream 9 that is withdrawn from the secondary separation vessel 7. In this configuration the third stage mixing tank 10 recombines the coarse solids, water and additional solvent and redisperses the asphaltene agglomerates that are fed to the third stage asphaltene separator vessel 11. Asphaltene agglomerates, coarse sand and water underflow stream 12 are fed into stripping unit 17 in which either fuel gas or steam and vacuum are used to remove the residual solvent from the solids and waste stream 18. Recovered solvent is combined with the overflow steam of the third stage separator vessel 11.

For a description of the other components depicted in the flow scheme of Fig. 2 reference is made to the detailed description with reference to Fig. 1.

In all embodiments of the method according to the invention that are depicted in the drawings the shear level generated in the agitating tank is crucial to the formation of asphaltene agglomerates in which the ultrafine clays are co-absorbed.

Example No. 1

5

10

15

20

25

30

The following procedure was followed in an initial laboratory bench test of the method according to the invention.

A sample of bitumen froth underflow was obtained from a froth treating pilot plant and put in beaker. The volume of bitumen froth underflow sample was 500 ml. The composition of the bitumen froth underflow sample was as follows: bitumen - 15.2% weight, asphaltenes content of bitumen - 5.2% of weight of sample, solvent - 30.4% weight, solids - 10.9% weight and water - 43.5% weight.

5

10

15

20

25

The bitumen troth underflow sample was stirred with an air driven impeller rotating at the speed of 500 rotations per minute for ten minutes. The power rate at which the impeller was driven was about 3 kW per m³ of volume of the beaker. The speed of rotation of the impeller was such to impart sufficient shear to the mixture to encourage formation of agglomerates of asphaltenes and co-absorption of the asphaltenes with the very fine clays but not so much shear as to cause the agglomerated asphaltenes to redisperse. The mixture was then allowed to settle and a middle layer of asphaltenes began to form. This middle layer was stirred gently which encouraged it to release water and solids into a lower layer, and hydrocarbons and solvents into an upper layer. These layers were removed and their respective volumes and compositions were measured or deduced to be as follows:

Upper layer: Volume = 50 to 75 ml.

Composition = bitumen - 33.3% weight,
asphaltenes content of bitumen in upper layer 3.7% of weight of upper layer, solvent - 66.6% weight,
solids - 0% weight and water - 0.1% weight.

Middlings layer: Volume = 200 ml.

Composition = bitumen - 17.1% weight,
asphaltenes content of bitumen in middlings layer 13.7% of weight of middlings layer, solvent 34.4% weight, solids - 14.3% weight and
water - 34.2% weight.

Lower layer: Volume = 250 to 275 ml.

Composition = bitumen - 1.5 weight, asphaltenes
O% weight, solvent - 1.5 weight, solids - 16.6% weight and
water - 83.3% weight.

The results described above were unexpected, as follows. The upper layer was virtually devoid of water and solids and the lower layer was virtually devoid of hydrocarbon. The middlings layer contained a significant proportion of the asphaltenes present in the treated sample. These asphaltenes can be removed and handled as a separate stream. Further, the middlings stream contains the very fine clays in the asphaltene agglomerates.

Removal of these very fine clays in this fashion makes subsequent handling of the lower layer and disposal of solids less troublesome.

Example No. 2

5

10

15

20

25

30

The following procedure was demonstrated in a laboratory scale pilot plant test of the method according to the invention.

The laboratory pilot was configured according to the flow scheme of Fig. 2. Bitumen froth obtained from a froth treating pilot plant and recycle solvent were combined to achieve a solvent to bitumen ratio of 1.6 by weight. The mixture was agitated in a first mixing vessel 3 with a power input of between 0.5 and 5 kW per $\ensuremath{\text{m}}^3$ of volume and introduced into a settling vessel 4. Underflow was withdrawn at a rate sufficient to remove all the water, solids and agglomerates to achieve a clean hydrocarbon product. This stream was combined with a solvent rich stream 22 from vessel 11 resulting in a solvent to bitumen ratio of about 10 and was introduced into mixing vessel 6. The contents of vessel 6 were agitated with a power input between 0.5 and 5 kW per $\ensuremath{\text{m}^3}$ of volume and introduced into separation vessel 7. Middlings stream 9 and underflow stream 8 were withdrawn from the gently agitated vessel 7 using between 0.1 and $0.5\ \text{kW}\ \text{per}\ \text{m}^3$ of volume. These two streams were combined

with fresh solvent to achieve a solvent to bitumen ratio of about 20 and introduced into mixer 10 which was agitated with a power input of between 0.5 and 5 kW per m³ of volume. Settler 11 yielded a clean hydrocarbon product which was recycled to join stream 5. The underflow stream 12 was collected for residual solvent recovery. Clean solvent and bitumen stream 13 was sent to tankage 23 and solvent recovery 24. The following table provides a summary of the overall bitumen material balance and asphaltene rejection.

5

10

	Total	Assay &	Bitumen	Distri-
	Mass (g)	Bitumen	Mass (g)	tion
Input to				
Pilot				
Test:				
Froth Feed	€84.0	55%	377	100%
Sclvent	979.0			····
Products from				
Pilot Test:				
Final				
Underflow	539.0	0.1%	3	0.8%
Final	936.1	38.0%	356	99.2%
Overflow			÷	
Solvent Loss	87.9			

Asphaltene rejection from the bitumen in the froth feed is given in the following table

	% Asphaltenes in	% Ash in Bitumen
	Bitumen	
Froth Feed	15.5	0.6
Product	10.1	0.05

illustrating a reduction of 26% of the asphaltenes in the feed and 92% reduction of ash from the bitumen in the froth.

The results of the laboratory pilot plant operation confirm the laboratory bench data which illustrates the performance of the asphaltene agglomeration process according to the invention.

5

CLAIMS

- 1. A method for processing an oil sand froth containing hydrocarbons, water, clays and coarse solids that is withdrawn as an underflow stream from a lower part of a primary fluid separation versel of a paraffinic solvent froth treatment unit in an oil sand processing plant, the method comprising the steps of:
- agitating the underflow stream at such a shear level that agglomerates of asphaltenes and clays are formed whilst dispersion of the thus formed agglomerates remains limited;
- adding paraffinic solvent to remove entrained bitumen;

5

10

15

20

- separating at least a substantial portion of said agglomerates from the other components of the underflow stream; and
- -- withdrawing the thus separated agglomerates as a separate product stream.
- 2. The method of claim 1, wherein the underflow stream is agitated by an impeller which is located in an agitating tank.
- 3. The method of claim 2, wherein the impeller is driven at a power rate which is between 0.5 and 5 kW per m^3 of the volume of the agitating tank.
- 4. The method of claim 2 or 3, wherein the agitated
 underflow stream flowing from the agitating tank has
 solvent added to it and is fed into a secondary
 separation vessel in which the fluid mixture is agitated
 at a lower shear rate than in the agitating tank, which
 lower shear rate is low enough to maintain the

agglomerates of asphaltenes and clays within an intermediate fluid layer but high enough to release other hydrocarbons into an upper fluid layer and to release water and coarse solids into a lower fluid layer within said vessel and which vessel is equipped with a lower fluid outlet through which at least a substantial proportion of water and coarse solids are removed from the vessel, an intermediate fluid outlet through which at least a substantial proportion of said agglomerates of asphaltenes and clays are withdrawn from the vessel and an upper fluid outlet through which at least a substantial proportion of other hydrocarbons are removed from the vessel.

- 5. The method of claim 4, wherein the underflow stream is gently agitated in the secondary separation vessel by means of a stirring device which is slowly rotated about a substantially vertical axis of rotation and which is equipped with a number of rakes and/or pickets.
- 6. The method of claim 5, wherein the stirring device is driven at a power rate which is between 0.1 and 0.5 kW per m³ of volume of the secondary separation vessel.

Smert & Digger Ottowa, Conada Petent Agains

Ġ

10

15

20

